Titanium Sputtering Target And Manufacturing Method of the Same

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ABSTRACT

Objective

Titanium target and the manufacturing method of the same are provided for improved step coverage and reduced particle numbers at contact holes of high aspect ratios.

Content

This invention provides a titanium sputtering target having (1) the X-ray intensity ratios: (0002)/(10-11)≥0.8, and (0002)/(10-10)≥6 at the sputtering surface; (2) the average grain diameter less than 20 μm; (3) the area fraction of grains with needle-like structure less than 20%; (4) completely recrystallized structure. The dispersion range of the grain diameter is ideally within 5-15μm. This type of target is manufactured by more than 75% cold work of titanium plate followed by annealing in the temperature range of 300-480°C for the control of recrystallization structure.

Claims

1. Titanium sputtering targets with characteristics of X-ray diffraction intensity ratio at the sputtering surface being (0002)/(10-11)≥0.8 and (0002)/(10-10)≥6, and the grain size of rescrystallized structure being less than 20 μm.

2. Titanium sputtering targets with a texture corresponding to X-ray diffraction intensity ratio of (0002)/(10-11)≥0.8 and (0002)/(10-10)≥6 at the sputtering surface, average grain diameter less than 20 μm, and less than 20% area fraction of needle-like structure.

3. Titanium sputtering targets according to Claims 1 or 2 where the oxygen is less than 600 ppm.

4. Titanium sputtering targets according to Claim 1 through Claim 3 where the dispersion range of grain size in the recrystallized structure is 5-15 μm.

5. The manufacturing method for Ti sputtering target comprising more than 75% accumulated deformation of cold work followed by annealing at 300-400°C to produce Ti sputtering target with characteristics of X-ray diffraction intensity ratio at the sputtering surface being (0002)/(10-11)≥0.8 and (0002)/(10-10)≥6, and average grain size being less than 20 μm in the recrystallized structure.

The Details of Invention

Areas of Industrial Application

This invention provides Ti target used for forming Ti film by sputtering and the manufacturing method of the Ti target.

Conventional Technology

Recently, LSI requires higher and higher density of micro-interconnects due to the demand of increased integration, increased function, and higher reliability. As a result, the contact holes/vias in the interconnects become narrower, and the doped P-type and N-type layers also have to be formed in narrower areas. If interconnects such as Al are formed directly on Si substrate with shallow doping layer, interdiffusion between Al and Si occurs. Once the Al diffuses through the doped layer, the structure of semiconductor is destroyed. To prevent the interdiffusion, a layer of refractory material such as MoSi, WSi or WTi is formed between the interconnects and the Si substrate.

TiN has recently been used as a barrier layer which has excellent ability in preventing the occurrence of interdiffusion. For the commercial application of TiN, studies have been performed to decrease the contact resistance between the TiN compound layer and the substrate. For example, one effective way to decrease the contact resistance between the Si substrate and the TiN layer is to form a TiSi compound layer between the TiN layer

and the Si substrate as shown in Semiconductor World 1992,12, pp196-205, or Semiconductor World 1989, 12, pp189-192).

TiSi compound can form by two ways: (1) as shown in the above references, Ti film is formed on Si substrate by sputtering Ti target followed by a heat treatment in an atmosphere of N_2 or NH_3. At the same time of TiN formation during the nitriding process of Ti, TiSi compound also is formed due to the reaction between pure Ti film and Si substrate; (2) TiSi compound is formed after heat treating the very thin Ti film deposited on the wafer by sputtering Ti target. After the TiSi layer is formed, a TiN film of certain chemical stoichiometry is deposited on TiSi layer through reactive sputtering of Ti in nigtrogen.

As mentioned before, Ti targets are used often for sputtering deposition of TiN or TiSi film for highly integrated LSI. Different methods to improve the quality of Ti target had been suggested. For example, as described in Japanese Patent 75301, resistance of film is reduced by reducing the concentration of oxygen. Also target with high purity eliminated the influence of radioactive elements.

In addition, as described in Japanese Patent 214521, film forming rate is increased by using Ti targets with (0002) texture. In Japanese Patent 10107 or 280009, a processing method for obtaining fine structure of recrystallization is described. This method comprises control of cold work and heat treatment. The fine structure of recrystallization supresses particale formation and results in more uniform film thickness distribution.

Issue Solved by This Invention

As the level of integration of LSI becomes higher, the contact holes have a tendency to become narrower and shallower. It is thus difficult to form a uniform film at the bottom of the contact hole by sputtering. If the atoms sputtered off the target by Ar ion have a perpendicular incidence relative to the wafer, there is no problem in depositing a film at the bottom of the hole. In fact, there is a distribution of emission directions for the atoms bombarded off the target by sputtering. It thus means that some atoms arrive at contact holes with inclined incidence. The more the atoms with inclined incidence are, the thinner the film at the bottom is and the thicker the film on the side-wall of the contact hole is. Therefore, the opening of the contact hole becomes smaller during sputtering which makes it more difficult to form a uniform film at the bottom.

For solving this problem, as mentioned in Electronic Journal 1944, 10, p132, the following methods have been used in which the film is formed only by the atoms with perpendicular incidence to the substrate. (1) A plate called collimator is inserted between the target and the substrate. However, most of the sputtered atoms were deposited on the collimator in this method. Therefore film formation rate and productivity is decreased significantly. In addition, if the Ti film adhered on the collimator dilapidates and falls down to the substrate, it may cause short circuit or open circuit. Due to these concerns, the commerciallization of this technique is slow. (2) Increasing the distance between the

target and the substrate is another method to permit only the atoms with perpendicular incidence to the substrate to reach the substrate surface. However, the film formation rate of this method decreases significantly and so does the productivity. In addition, it requires more vacuum room for sputtering, and therefore the instrument becomes bigger and higher cost.

As contact holes become smaller, particles generated become more serious defects. The structure with fine recrystallized grains is an affective way to prevent the generation of particles, which has been recorded in Japanese Patent 10107 or 280009. It is not enough to have fine grains for the purpose for reducing the generation of particles. Patent 214521 proposed target having (0002) texture to increase the film formation rate. The investigation of this invention indicated (0002) texture alone cannot suppress the generation of the particles effectively. The objective of this invention is to provide Ti target and its manufacturing method to achieve the following: more focused atomic emssion in sputtering so that it is easier to deposite films in narrower and deeper contact holes, and also to reduce generation of particles.

Approach for solving the issue

The inventors investigated the relationship between the structure of titanium and the direction of sputtering atoms and the relationship between the structure of titanium and the generation of particles. The cold work ratio applied to the target was larger than that in the conventional cold work process and the heat treatment temperature used was lower than that in the conventional process. As a result, the sputter surface of the Ti target has strong crystallographic orientation of (0002) as opposed to random orientation. The target has a strong texture with reduced orientation intensity of (10-10) and (10-11). In addition, a recrystallized structure with very fine grain size is obtained. The cold work ratio mentioned in this invention represents the accumulative cold work ratio that should be applied to the target. By uing this kind of Ti target in sputtering, both goals of decreasing particles and increasing unidirectional emission of sputtered atoms are accomplished.

This invention provides Ti targets having X-ray diffraction intensity ratios of (0002) / (10-11)≥0.8 and (0002) / (10-10) ≥ 6 from the sputter surface, average grain size of less than 20 μm, and average grain size of less than 10 μm under ideal condition. The negative sign for the index in the parenthes of a given crystal plane represents a direction negative to the crystal axis. The invented Ti target is made by cold work of more than 75% on Ti plates followed by heat treatment at 300-400°C to give X-ray intensity ratios of (0002) / (10-11) ≥ 0.8 and (0002) / (10-10) ≥ 6 at the sputter surface, and mean grain size less than 20 μm. The more desired orientation of grains is stronger (0002) and weaker (10-11), i.e. (0002) / (10-11) ≥ 1.0.

The method used in manufacturing Ti target in this invention is characterized by high cold work ratios that conventional methods can not achieve, and by much lower heat treatment temperature than the conventional methods. The upper limit of the ratio for the conventional cold-work on Ti targets is 70%. Cracks can develop easily at the edges of

the material if the cold work ratio was more than 70%. In addition, m re than 70% cold work ratio imposes too much load on rolling machine. Therefore, cold work ratio of more than 70% is not usually applied. This invention is distinguished from the conventional process in that cold work ratio of more than 75% is adopted. Higher cold work ratio is achieved, for example, by applying multiple rolling passes with 15% work ratio in each rolling pass and simultaneously adjusting the shape of the Ti plate to obtain a round edge.

Another advantage of low temperature annealing to obtain a recrystallized structure is to prevent deteriorated performance of Ti target due to existence of needle-like structure. Needle-like structure is an indication of heat treatment in β phase or twinning, which is not desired.

The area proportion of needle-like structure in this invention is not more than 20%. Needle-like structure which has hcp crystal structure forms while β phase is transformed to α phase during cooling from high temperature such as 800°C or higher. Twins form if the material is rapidly cooled from a temperature higher than 500°C. In this invention, grains recrystallize at low temperature and therefore neeedle-like structure formation during cooling is suppressed.

Low oxygen concentration is desirable for reduced resistance of Ti film. Thus, it is desired to choose row material with oxygen concentration less than 600 ppm. If the distribution of grain size is wide, the sputtering erosion of Ti target can not be uniform. Therefore, a microstructure with more uniform grain size is desired. Due to adopting higher cold work ratio to give a large deformation and lower temperature for recrystallization, the grains size obtained in this invention become finer and more uniform. The dispersion of grain size can be kept at a range of 5-15 μm.

Effect of the Invention

As mentioned above, the most important feature of the invention is that the sputter surface has a texture of (0002) characterized by X-ray diffraction intensity ratios of (0002) / (10-11) ≥ 0.8 and (0002) / (10-10) ≥ 6 instead of random orientation, and fine recrystallized microstructure. The (0002) crystal plane is the close-packed plane in hcp structure. The recrystallization during the heat treatment following the cold work with a work ratio less than 70% does not result in strong (0002) texture, and instead results in random crystalline orientation distribution. It is usually considered that about 500°C is a suitable recrystallization temperature for the refining of grain size. But, the strong (0002) texture that has (0002) / (10-11) ≥ 0.8 and (0002) /(10-10) ≥ 6 can not be obtained under such condition.

In this invention, the cold work deformation of Ti plate is more than 75% followed by recrystallization at lower temperature. Therefore, a microstructure of recrystallization with mean grain size less than 20 μm is obtained. Fine recrystallized microstructure without fiber-like structure can form at a temperature lower than 480° C. Since the work ratio is high, the (0002) texture created in the cold work can be kept after the heat

treatment. Therefore, the Ti target has (0002) texture as well as fine grain microstructure formed by the low temperature recrystallization mentioned above.

The sputter surface of the Ti target in this invention has increased atomic density due to strong texture of (0002) which is the close-packed crystal plane in hcp crystals. Also the intensity of (10-11) and (10-10) texture are reduced. Atoms emitting from the sputtering surface are influenced by the neighbor atoms and it is difficult for the sputtered atoms to fly in a direction inclined to the surface. Therefore, there is high proportion of atomic emission perpendicular to the sputter surface if there is a high proportion of close-packed (0002) plane at the sputter surface. Hence, the atoms arriving at contact holes perpendicularly from this type of targets are more than those from a target with a random crystalline orientation.

The grains of Ti targets manufactured by vacuum melting or electron beam melting have random crystalline orientations. The dominant X-ray diffraction intensity is from (10-11) crystal plane. According to the ASTM card, the X-ray diffraction intensity ratio for (0002)/(10-11) is about 0.2-0.3 from a sample with a random grain orientation distribution. It is desirable that the sputter surface does not comprise low density crystalline planes.

Conventional technique only considered to have (0002) orientation. The experimental results of this invention showed that decrease of low density crystal planes has significant effect on the texture uniformity of the target. The characteristics of the invention are close-packed atomic plane (0002) on the sputter surface and controled ratio of non-close-packed atomic planes (10-11) and (10-10). Therefore, the ratio (0002) / (10-10) $\geq$ 6 is required according to the above point of views instead of (0002) / (10-10) $\geq$ 0.8. As a result, it becomes possiblt to obtain unidirectional emission of sputtered atoms.

In the invention, to keep the crystalline orientation as mentioned above is very important for suppressing the generation of particles. The strong texture allows the sputter surface to approach one that is composed of the same crystal plane. The difference of erosion rate of a target can be reduced if the grains have very similar orientations. In this invention, large steps at the grain boundaries between similar grains on the sputtered surface cannot occur easily due to the strong texture in this invention by controling any one of (0002), (10-11), or (1010). Consequently, the generation of particles is reduced by preventing the abnormal discharge of electricity induced at the large elevation steps of grain boundaries.

In the invention, for decreasing the generation of particles, it is necessary to refine the recrystallization structure, that is mean grain size less than 20 μm, as well as to control the grain orientations. If it were only for the purpose of having a strong crystallographic texture, the fiber structure obtained from the cold working is good enough. However, the strong heterogeneity in this structure and the residual strain of cold working result in non-uniform erosion of the target during sputtering and thus increased generation of particles. Once the target has a recrystallized structure, the generation of particles can be reduced

due to the decrease of heterogeneity and the removal of the large amount of strain from the cold working.

It is very important that the size of recrystallized grains is less than 20 μm, preferably smaller than 10 μm. In the conventional technology, recrystallization takes place at a temperature higher than 500°C. As a result, coarse grains of 30-40 μm are obtained. This is one of the reasons for the increased particles generation. In this invention, the recrystallization at a lower temperature becomes possible due to the large ratio of accumulative cold-working. Because the average grain size is less than 20 μm, the generation of particles can be prevented effectively.

The Ti target obtained by this invention has fine grains and a strong texture. The elevation difference between grains does not increase even though the erosion rate of different grains are different during the sputtering. Therefore, the increase of particle generation is prevented effectively. The more the dispersive of the grain size, the easier the formation of the grain boundary steps is. Thus, it is desired that the dispersion of grain size is small. In this invention, the ideal dispersion of grain size is 5-15 μm.

For getting a stable sputtered film, it is desired to remove the worked layer and oxide film on the target surface to obtain a stable sputtering surface by pre-sputtering (also called dummy sputtering).The Ti target manufactured by this invention only needs a short period of pre-sputtering since it has strong orientation and fine grains. It is desirable to have as short pre-sputtering time as possible in fabrication. The pre-sputtering time is the time spent for removing the grains in the worked layer and oxidized layer. Since a stable sputter surface can be obtained in a short period of time due to strong texture and refined grains, the pre-sputtering is shortened. In addition, if twins exist in the recrystallized grains or needle-like structure exists after cooling from β-Ti phase, needle-like concave and convex appear within a grain due to sputter erosion. This surface topography results in the increase of particle generation. It is desired that the area ratio of the grains with twins or needle-like structure is below 20%, preferrably below 5%.

As mentioned previously, the cold working deformation ratio is required to be more than 75% in this invention. The more than 75% cold work required in this invention is for the purpose of obtaining strong texture and fine recrystallized grains. Since a high cold work ratio is imposed, the recrystallization can occur at low temperature and hence very fine recrystallized grains can be obtained. The recrystallization at a low temperature can also suppress the formation of needle-like structure that causes the generation of particles. In this invention, the heat treatment temperature for recrystallizing is 300-480°C, preferably 350-450°C. It is proved that annealing temperature higher than 480°C following more than 75% work ratio causes grain coarsening and occurrence of needle-like structure.

Examples

Example No.1

A Ti ing t of 5N grade (99.999% purity) was cold rolled with a cumulative defamation of 78% after forging. The structure of ingot, the structure after forging and cold rolling were investigated with an optical microscope so that the changes of structure during the processing is analylized. Boiled nitric acid was used as etchant for sample preparation. The results are shown in Figs.5-7. As shown in the figures, there are structures where grain boundaries cannot be recognized in the processing from ingot to the rolled product. The Ti targets of this invention and the targets for comparison were cold rolled and then heat treated at different temperatures in a range of 280-580°C. The oxygen contents of all the targets are in a range of 400-500 ppm.

The structures of the heat-treated targets were observed with the method mentioned above. Fig. 1 shows the typical structure of this invention after heat treating at 400°C for 6 hours. It can be seen from Fig.1 that the Ti target of this invention comprises fine grains and no deformation structure. Fig.2 shows the structure for the same processing condition as that in Fig.1 except for heating to 470°C. Larger grain size and small amount of needle-like structure can be seen in Fig. 2 compared to Fig. 1.

Fig.3 shows the structure for the same condition as that in Fig.1 except that the heat-treatment temperature was controled at 280°C. It is observed that the fine structure same as shown in Fig.2 begins to appear. Recrystallization has definately started in this sample, but majority of it remain as deformation structure. As with the Ti target to be mentioned later, a great quantity of particles will occur in this condition, which is not desired. Fig.4 shows the structure for the same processing condition except for heat treatment at 580°C. Compared to Fig.1 and 2, it has coarse grains and needle-like structure in all grains.

The intensity ratio of (0002) to (10-11) and (10-10) at the sputtering surface of all these targets were measured using X-ray diffraction. In addition, the maximum diameter of the grains, D_{max}, the minimum diameter of the grains, D_{min}, and the average diameter of grains, D_{av}, were measured on the microstructure pictures with magnification of 400X by the interception method. At the same time, the area fraction of needle-like structure was measured. The results are shown in Table 1. The grain size of the sample treated at 280°C was not recorded in Table 1 because grain size can measured with the existence of the residual rolling structure.

Table 1

No.	Heat Treatment Temp. °C	Diffraction Intensity Ratio (0002)/(10-11)	(0002)/(10-10)	Grain Diameter D_{min}	D_{max}	D_{av}	% Needle-Like Structure	Annotation
1	400	1.48	11.2	3	12	9	0	This Invention
2	470	1.18	8.3	5	13	11	8	This Invention
3	280	1.85	20.0	-	-	-	-	Comparison
4	580	0.75	4.0	32	63	49	99	Comparison

As shown in Table 1, the X-ray diffraction intensity ratio (0002)/(10-11) and (0002)/(10-10) for the Samples 1 and 2 provided by this invention are greater than 0.8 and 6, respectively. The sputter surfaces are composed of close-packed plane (0002) mainly. There is little fraction of the (10-10) plane which has the least atomic density, on the sputter surface. As shown in Figs.1 and 2, the sputter surface of the samples provided by this invention is composed of fine recrystallized grains without the rolling structure as well as (0002) strong texture. Especially, the sample heat treated at 400°C has mean diameter of grains smaller than 10 μm without any needle-like structure and the grain size dispersion is within a range of 5-15 μm.

The Ti film deposited by using these Ti targets were evaluated. The vacuum of the sputter chamber was 4×10^{-5} Pa, and the temperature of wafer was 250°C. Ar was used as sputtering gas with a pressure of 0.13 Pa. The sputtering power density was 12 w/cm^2. Thin films of 300 nm thickness was deposited on 4 inche wafers. Numbers of particles with size larger than 0.2 μm were measured. In addition, contact holes of 1 μm in diameter was formed by oxidizing the Si substrate. Ti film was sputtered onto the wafer under the above condition. The cross-sections of the wafers was created by cleavage and was investigated by an electron microscope. The thickness of Ti film at the bottom and the side wall of the contact hole were measured by the electron microscope. The film thickness at the bottom of the contact holes is labled as B and that on the side wall is labled as S. A high value of B/S means a large amount of sputtered atoms deposited at the bottom. The results are shown in Table 2.

Table 2

No.	Particles/Wafer	Bottom Coverage/ Side Coverage B/S	Annotation
1	24	1.67	This invention
2	32	1.52	This invention
3	153	1.72	Comparison
4	77	1.20	Comparison

As shown in Table 2, the Sample 3 which has rolling structure provides high bottom coverage, but also provides a significant number of particles which is not desired. Sample 4 processed with the method of this invention and heat-treated with an increased temperature has lower fraction of (0002) plane, coarse grains, large amount of needle-like structure. This target also generated very large particles which is not desired. In contrast to the comparison samples, Samples 1 and 2 of this invention have refined grain size without residual rolling structure after recrystallization, and high proportion of the close-packed plane (0002) and very low proportion of the least packed plane (10-10) on the sputter surface, as a result of rolling ratios up to 78% and heat treatment temperatures below 480°C. The generation of particles was suppressed and thick film at the bottom of the contact holes was obtained simultaneously by using the method of this invention.

Example N . 2

A 5N purity Ti ingot were forged and cold rolled with rolling ratios in a range of 50-80% as shown in Table 3. In order to obtain the recrystallization structures shown in Tabl 3 after cold rolling, the samples were heat treated at different temperatures for 60 minutes. As in the Example No.1, the intensity ratios of (0002)/(10-11) and (0002)/(10-10) were measured on the target surface by X-ray diffraction. D_{min}, D_{max}, and D_{av} were measured by interception method on the microstructure pictures with magnification of 400X. The area fractions of needle-like structure were also measured. The results are shown in Table 3. As in the Example No.1, the generation of particles and the film thickness at the bottom and the side wall of contact holes were evaluated after sputtering. The Sample 5 is the same material as the Sample 1. The oxygen contents of all examples are 400-500 ppm (Table 3).

Table 3

No.	Rolling Ratio	Heat Treatment temperature °C	Diffraction Intensity Ratio (0002)/(10-11)	(0002)/(10-10)	Diameter of grain (μm) D_{min}	D_{max}	D_{av}	Needle-Like Structure (%)	Annotation
5	78	400	1.48	11.1	3	12	9	0	This invention
6	85	375	1.54	11.6	3	11	8	0	This invention
7	50	520	0.76	4.8	15	300	180	30	Comparison
8	70	500	0.98	5.4	7	30	19	12	Comparison

Table 4

No.	Particles/Wafer	B/S	Annotation
5	24	1.67	This invention
6	25	1.69	This invention
7	58	1.24	Comparison
8	33	1.30	Comparison

As shown in Table 3, the Samples 7 and 8 for comparison have large grains and high proportion of needle-like structure due to low rolling ratio and high heat treatment temperatures for recrystallization. Compared to the Samples 5 and 6 of this invention, the Samples 7 and 8 have lower X-ray diffraction intensity of (0002) plane and higher intensity of least packed plane (10-10). As shown in Table 4, these targets generate more particles and provide thinner bottom coverage, which is not desired, compared to the Ti targets of this invention.

In order to determine the origins of particle generation, surface roughness was measured on the Sample 5 (this invention) and the Sample 7 (for comparison) after sputtering. The results of surface roughness measurements are shown in Figs. 8 and 9. The comparison between Figs. 8 and 9 shows that the target of this invention has lower surface roughness even though the target of this invention and the target for comparison have almost the

same grain size. This is because the grains in the target of this inventi n are oriented to a similar direction (strong texture). This is an indication that the difference in sputtering rate of different grains is not significant in the target of this invention. Therefore, the target of this invention can prevent the increase of particle generation even in the late stage of sputtering compared to the comparison targets.

Example No.3

The Ti target Sample 5 (this invention) and the Sample 7 (comparison) in the Example No. 2 group were used to deposit TiN films by reactive sputtering. The chamber vacuum was 4×10^{-5} Pa. A mixed gas of Ar and N_2 (50:50) were used for the reactive sputtering at a pressure of 0.26 Pa. The temperature of the wafers was 250°C and sputtering power density was 15 w/ cm^2. TiN films of 300 nm thickness were formed on 4 inch wafers. The numbers of particles larger than 0.2 μm were measured. As in the Example No. 2, the film thickness at the bottom and the side wall of the contact holes were measured. The thickness of film at bottom is labled as B and that on the side wall is labled as S. The ratio of B/S was calculated. The larger the value of B/S is, the more the sputtered atoms are deposited to the bottom. The surface roughness, R_{max}, was measured and evaluated after sputtering. As shown in Table 5, the results for the invented target is represented by No. 9 and the results for the comparison target is represented by No. 10.

Table 5

No.	Particles/ Wafer	B / S	R_{max} (μm)	Annotation
9	152	1.32	2.5	This invention
10	326	1.08	15.2	Comparison

As in the Example No. 2, the target of this invention provides fewer particles and thicker bottom coverage than the comparison target in the reactive sputtering. The surface roughness is also smaller on the target of this invention than the target of comparison in the condition of reactive sputtering. Due to the special crystalline orientation and the fine recrystallized grains, the differences of sputtering rates among different grains are small, which is effective in preventing the increase of particle generation even in the late stage of sputtering.

The Effect of the Invention

By using the Ti targets of this invention, Ti film can be deposited easily in narrow and deep contact holes during regular sputtering or reactive sputtering. The generation of particles is reduced with the invented targets not only at the early stage of sputtering, but also in the late stage of sputtering. A stable sputtering surface can be established after a short period of time since the invented targets have fine grains with a strong texture. Therefore, this invention provides an effective target for depositing films as a part of the semiconductor devices which becomes higher and higher densities in recent years.

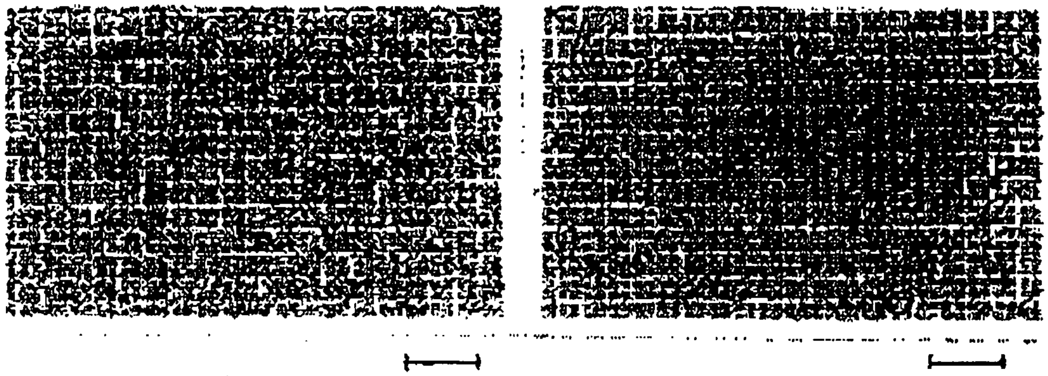
Captions

Fig.1 The microstructure picture of the invented target heat treated at 400°C.
Fig.2 The microstructure picture of the invented target heat treated at 470°C.
Fig.3 The microstructure picture f the comparison target heat treated at 300°C.
Fig.4 The microstructure picture of the comparison target heat treated at 500°C.
Fig.5 The microstructure picture of the ingot.
Fig.6 The microstructure picture after forging.
Fig.7 The microstructure picture after rolling
Fig.8 Surface roughness on the invented target after sputtering.
Fig.9 Surface roughness on the comparison target after sputtering.

【図1】

図面代用写真

加熱温度 400℃ (本発明例)

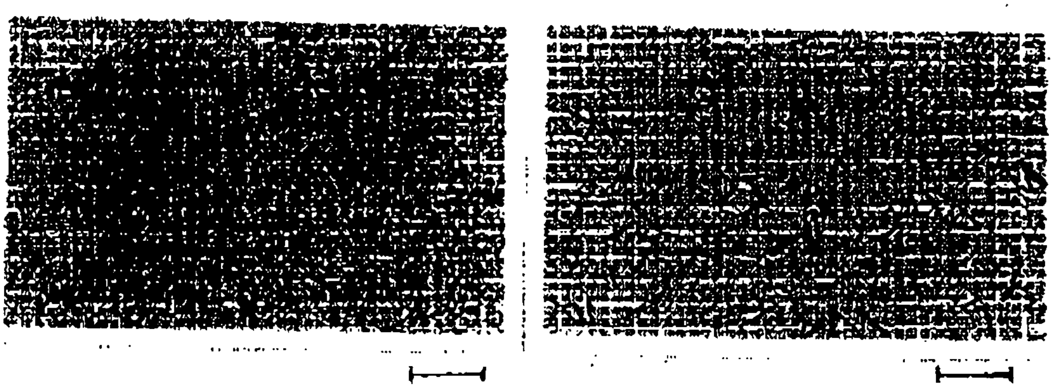


(×100) 100μm (×400) 25μm

【図2】

図面代用写真

加熱温度 470℃ (本発明例)

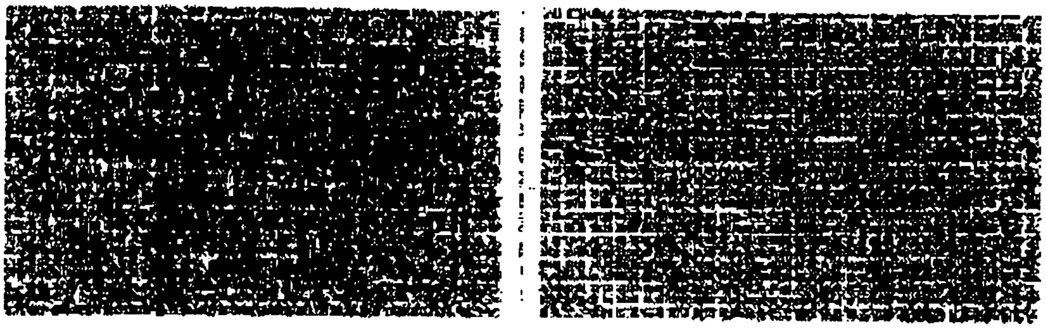


(×100) 100μm (×400) 25μm

【図3】

図面代用写真

加熱温度 300℃（比 較 例）

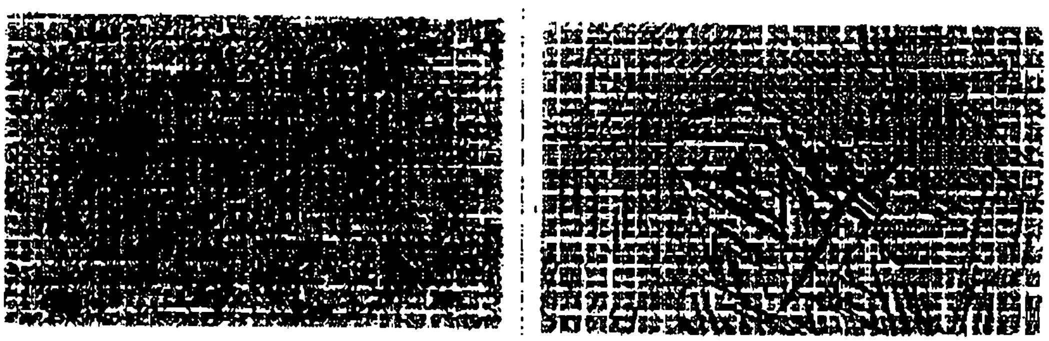


（×100） 100μm （×400） 25μm

【図4】

図面代用写真

加熱温度 580℃（比 較 例）

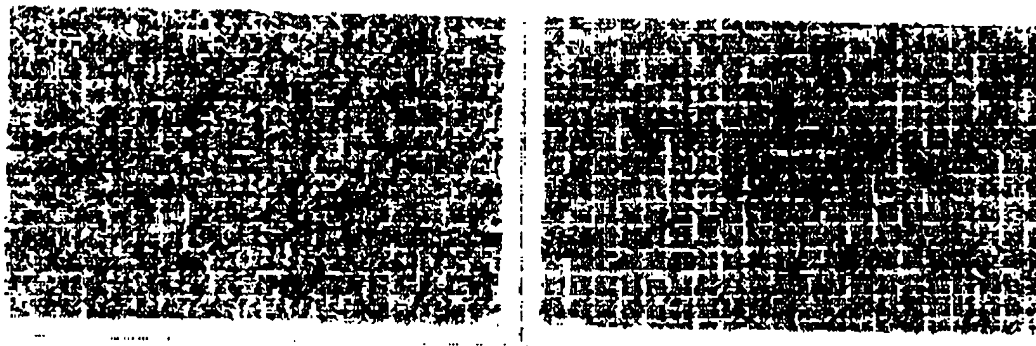


（×100） 100μm （×400） 25μm

【図5】

図面代用写真

インゴット

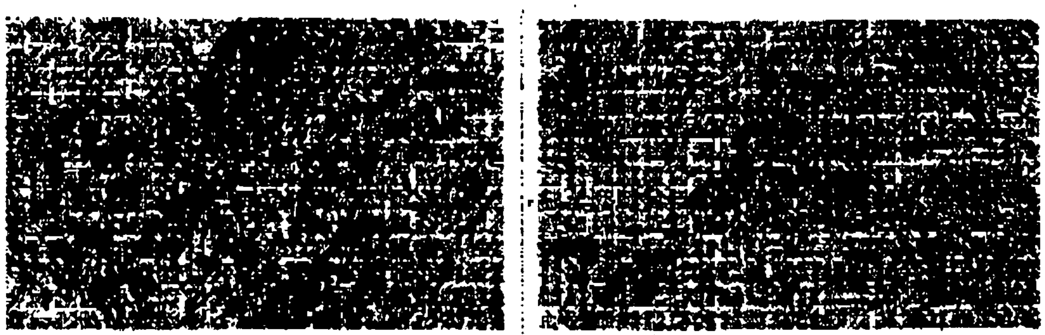


(×100) 100μm (×400) 25μm

【図6】

図面代用写真

鍛 造

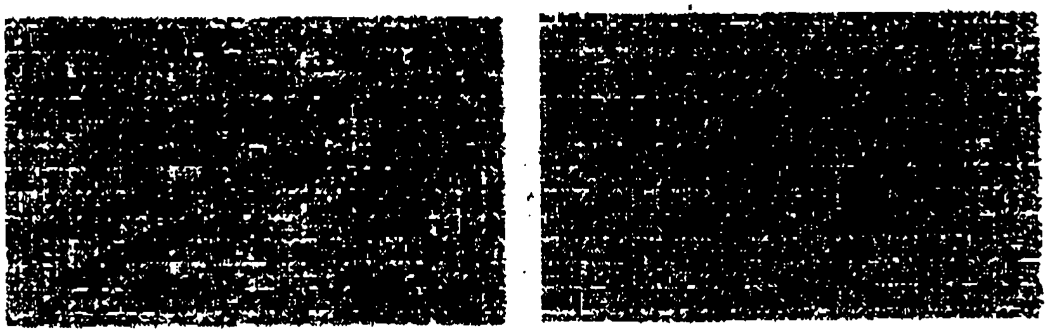


(×100) 100μm (×400) 25μm

【図7】

図面代用写真

圧　延



(×100) 100μm　　　　(×400) 25μm